Mo/B₄C/Si multilayer-coated photodiode with polarization sensitivity at an extreme-ultraviolet wavelength of 13.5 nm

Benjawan Kjornrattanawanich, Saša Bajt, and John F. Seely

A silicon photodiode coated with an interface-engineered Mo/Si multilayer was developed as a polarization sensitive detector. The Mo/B₄C/Si multilayer was designed to reflect 13.5-nm extreme-ultraviolet (EUV) radiation at an incident angle of 45°, at which the maximum polarization sensitivity occurs. The sensitivity of this specially coated photodiode and its polarization responses were determined by measurement of the reflectance and transmittance of the multilayer coating with synchrotron radiation. The Mo/B₄C/Si multilayer was found to reflect 69.9% of the s-polarized radiation and only 2.4% of the p-polarized radiation, thus transmitting approximately 0.2% s-polarized radiation and 8.4% p-polarized radiation at a 13.5-nm wavelength and a 45° angle of incidence. A polarization ratio, $(T_p - T_s)/(T_p +$ T_s), of 95% was achieved with sufficiently high sensitivity from this photodiode. This result demonstrates the high polarization sensitivity and the usefulness of multilayer-coated photodiodes as novel EUV polarimeters. © 2004 Optical Society of America

> effective reflectors for EUV radiation. The ability to reflect narrow spectral bandpasses at near-normal

> incidence has made multilayers the coatings of choice

for focusing optics employed in present solar tele-

scopes, such as the Extreme Ultraviolet Telescope³

and the Transition Region and Coronal Explorer,⁴ as

well as in future solar spectrometers, such as the

Solar-Extreme Ultraviolet Imaging Spectrometer.⁵

In addition to providing high normal-incidence reflec-

tance, the peak wavelength (λ) of a multilayer is tun-

able with its period thickness (d) and with the

incident angle of radiation (θ) in accordance with the

Bragg condition $\lambda = 2d \cos \theta$ (ignoring the refraction

correction). To be used as a polarizer, the multilayer

period thickness can be chosen so that its reflected

peak wavelength occurs near the Brewster angle.

The Brewster angle is defined as the incident angle at

which the reflected beam from each interface becomes linearly polarized; that is, when the intensity

of the reflected rays with the electric field vector par-

allel to the plane of incidence reaches its minimum

for absorbing material or becomes zero for nonabsorb-

ing material. Once reflectance optimized around its

Brewster angle, a multilayer would strongly reflect

the s-polarized component, as well as transmit the

p-polarized component, within the restricted wave

band established by the multilayer coating. An op-

tical system that is equipped with a monolithic trans-

mittance polarizer and an integrated detector

OCIS codes: 230.5440, 230.4170, 230.5170, 260.5430, 260.7200.

1. Introduction

Silicon photodiodes are semiconductor radiation sensors that have been used widely as x-ray and extreme-ultraviolet (EUV) detectors. Applications of these detectors include solar study, EUV lithography, x-ray microscopy, and plasma physics. Besides high responsivity and good radiation hardness, many experiments require detectors with high polarization and wavelength selectivity. An accurate determination of the state of polarization is desirable for the study of polarized radiation from solar and astrophysical plasmas, which are intense sources of EUV radiation. This study will potentially lead to a better understanding of the magnetic field formation, particle acceleration process, and heating mechanism of the solar corona and flares.^{1,2}

Multilayer coatings have been demonstrated to be

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B. Kjornrattanawanich (benjawan@bnl.gov) is with the Universities Space Research Association, National Synchrotron Light Source, Beamline X24C, Brookhaven National Laboratory, Upton, New York 11973. S. Bajt is with the Lawrence Livermore National Laboratory, L-395, Livermore, California 94550. J. F. Seely is with the Naval Research Laboratory, Space Science Division, Washington, D.C. 20375.

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provides simple alignment and therefore is more compact and preferable in all applications to a system equipped with a reflectance polarizer and a separate detector. Successful fabrications of freestanding multilayers for use as transmittance polarizers and beam splitters have been demonstrated by several techniques similar to those used in microelectronic fabrications. ^{6–8} However, such processes are somewhat complicated and require good knowledge about a thin film's chemical, optical, and, most important, mechanical properties. High intrinsic stress (compressive or tensile) in multilayer films, for example, can cause wrinkles and eventually break the normally thin and delicate film substrates.

A novel feature of our device is the deposition of the multilayer coating directly onto a photodiode substrate. In this way, not only can the photodiode be used as polarization sensitive detector, but a complete characterization of reflectance, transmittance, and absorptance of a multilayer can be performed simultaneously without the need of a fragile free-standing sample. The gain in transmittance polarization can be increased by increasing the number of the multilayer periods. However, this benefit may be offset by an undesired increase in absorption through the whole stack of the multilayer and a resulting decrease in the signal from the underlying photodiode.

In the present study silicon photodiode detectors with multilayer interference coatings that provide polarization and wavelength selectivity at a wavelength of 13.5 nm are developed. Commercially available silicon photodiodes (AXUV-100G; from International Radiation Detector Incorporated) with a $10 \text{ mm} \times 10 \text{ mm}$ active area were used because they are known to provide high quantum efficiencies in this wavelength region.⁹ Briefly, the silicon p-njunction was formed between an epitaxially grown p-type region and an n-type defect-free region. An approximately 3–12-nm-thick SiO₂ layer was grown as a protective layer at the top. Owing to extensive studies of Mo/Si multilayers and their exceptionally high reflectance at a wavelength of 13.5 nm, a Mo/Si multilayer was selected as a coating for these diodes. The multilayers were optimized near the Brewster angle to obtain the highest transmittance polarization selectivity at 13.5 nm. The device's operating wavelength region can be shifted by selection of a different multilayer coating and optimization of the reflectance and transmittance properties at the desired wavelength.

2. Multilayer Fabrication

A typical multilayer mirror consists of a periodic stack of two alternating layers with different refractive indices. Because silicon is known to have relatively low absorption at wavelengths longer than the Si L absorption edge at 12.4 nm, it therefore has good optical contrast with molybdenum, resulting in high reflectance at a wavelength of ~ 13.5 nm. Nevertheless, a major source of imperfection that reduces the reflectance of Mo/Si multilayers is silicide formation

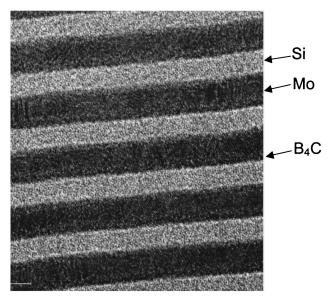


Fig. 1. Transmission electron microscopy cross-sectional image of a $\mathrm{Mo/B_4C/Si}$ multilayer. Molybdenum has a higher atomic number (Z=42) and therefore appears darker as it scatters more electrons than does silicon (Z=14). The transition layers that are due to silicide formation and that are normally observed at interfaces of $\mathrm{Mo/Si}$ multilayer (especially at the $\mathrm{Mo-on-Si}$ interfaces, which are thicker than the $\mathrm{Si-on-Mo}$ interfaces) can no longer be observed after the deposition of thin $\mathrm{B_4C}$ barrier layers at all $\mathrm{Mo-on-Si}$ interfaces.

at the interfaces between molybdenum and silicon layers. A low absorbing layer (boron carbide) was introduced as a barrier layer against silicide formation, and a reflectance as high as 70% at 13.5 nm was reported from the so-called interface-engineered Mo/Si multilayer.¹⁰ Because the silicide layer is much thicker at the Mo-on-Si interface than at the Si-on-Mo interface and because only a small reflectance improvement was found when these barrier layers were deposited at all the interfaces, approximately 0.4-nm-thick boron carbide barrier layers were deposited only at the Mo-on-Si interfaces for all the multilayers in this study. An improved interfacial structure can be seen from the cross-sectional transmission electron microscopy image of a Mo/ B₄C/Si multilayer that has relatively sharp, smooth interfaces with no sign of a transition region due to silicide formation at the Mo-on-Si interfaces (Fig. 1).

The multilayer coatings were fabricated with a dc-magnetron sputtering system at the Lawrence Livermore National Laboratory. An ultrahigh purity argon gas was used as a process gas with a process pressure of 1 mTorr. The base pressure of 2×10^{-7} Torr was normally achieved before deposition. The photodiode substrates were held facedown and spun on a platter, which rotated over the sputtering sources. The deposited thickness was computer controlled by variation of the rotational velocity of the platter.

During multilayer deposition, a mask with an 8-mm-round aperture covered the 10-mm-square active area of the photodiode. In addition, the perimeter of the active area was covered by a thin bead of nonconducting epoxy, which prevented the deposition material from coating the edge of the active area and adversely affecting the performance of the photodiode. These techniques were previously developed during the coating of photodiodes for the purpose of determining the optical properties of the coating.¹¹

For operation in the EUV region, the refractive indices of both molybdenum and silicon approach unity, and the Brewster angle was estimated to be 45°. The period thickness of the Mo/B₄C/Si multilayer was chosen to be 9.9 nm, with a ratio of molybdenum thickness to period thickness of 0.33 and 50 total periods. With this optimized coating, the s-polarized radiation reflected from all interfaces would constructively interfere at an incident angle of 45° and at an incident wavelength of 13.5 nm, and the p-polarized radiation would selectively transmit through the coating and generate a signal in the underlying photodiode. Molybdenum was coated as the first layer in the multilayer stack. An additional silicon layer of the same thickness as those within the multilayer was coated as a capping layer because silicon-capped multilayers are known to be very stable when exposed to EUV light. 12,13 The same multilayer coating was applied on a flat silicon wafer substrate and served as a witness coating. The multilayer period thickness of the witness sample was measured with a $Cu K_{\alpha}$ x-ray diffractometer and was determined to be 9.88 nm.

3. Polarization

A. Incident Radiation

The performance of the $\mathrm{Mo/B_4C/Si}$ multilayer-coated photodiode was determined by use of the Naval Research Laboratory's reflectometer at the National Synchrotron Light Source beamline X24C at the Brookhaven National Laboratory. This beamline uses a monochromator composed of a gold-coated mirror and a 600-line/mm diffraction grating that provides dispersed radiation in the spectral range of 2.8-110 nm (465-11 eV). The incident radiation obtained from the monochromator is polarized with the electric-field vector primarily in the plane of the synchrotron ring (in the horizontal direction). The degree of polarization of the incident radiation is defined as

$$\alpha = (I_H - I_V)/(I_H + I_V),$$
 (1)

where I_H and I_V are the intensities with the electric field vectors in the horizontal and vertical directions, respectively. The value of α can vary between +1 for $I_V=0$ and -1 for $I_H=0$. A previous study of the reflectance of a gold-coated flat mirror at an incident angle of 45° determined that the polarization α is approximately 0.83 (83%) for an incident wavelength of 20–22 nm at this beamline. Following the work cited in Ref. 14, the two grazing incidence mirrors associated with beamline X24C were replaced, and the polarization of the radiation propagating to the

reflectometer was measured during the present work by use of a similar method. The measured R_V and R_H (see definitions in Section 3.B) of the gold-coated mirror are shown in Fig. 2. Because the gold coating was thick (much larger than the incident wavelength) and no substrate-interference effect was observed in the reflectance spectra, the reflectance from the gold/ substrate interface was ignored in the fitting procedure. The fitted R_V and R_H values were calculated with the Fresnel formulas and with two fitting parameters: the degree of polarization of an incident radiation (α) and the surface roughness of the gold mirror (σ). The determined value of α was 0.89, and the σ value was 3.5 nm. The fitted value for the roughness was in good agreement with the values determined with atomic force microscopy on different samples. This is, indeed, a preferred method of determining the polarization of the incident radiation compared with the previously used method,14 where an assumption that $R_P \ll R_S$ at an incident angle of 45° is inaccurate for gold in the wavelength region of our interest.

B. Reflected and Transmitted Radiation

For a standard reflectometer setup at beamline X24C, the rotational axis of the sample-detector is in the vertical direction. The electric field vector of the incident radiation beam is primarily horizontal (and parallel to the plane of incidence), and the incident radiation is primarily p-polarized (see Fig. 3). The reflectance and transmittance of a multilayer can be measured versus the wavelength for different angles of incidence. The measurements for the case of primarily s-polarized radiation were achieved by rotation of the reflectometer 90° about the axis of the incident beam so the incident radiation was primarily s-polarized with the electric field vector perpendicular to the plane of incidence. In this orientation the rotational axis of the sample-detector is in the horizontal direction (and is normal to the incident beam).

In each reflectometer orientation the reflectance is equal to the ratio of the reflected intensity and the direct beam intensity (both were measured with the same reference photodiode). The measured reflectance in the vertical (R_V) and the horizontal (R_H) orientations can be expressed as

$$R_V = (R_P I_H + R_S I_V) / (I_H + I_V),$$
 (2)

$$R_H = (R_S I_H + R_P I_V) / (I_H + I_V), \tag{3}$$

where R_P are R_S are reflectances for 100% p-polarized and s-polarized incident radiation, respectively. R_P and R_S may be rewritten in terms of the measured R_V , R_H , and α as

$$R_P = [R_V(1 + \alpha) - R_H(1 - \alpha)]/2\alpha,$$
 (4)

$$R_S = [R_H(1+\alpha) - R_V(1-\alpha)]/2\alpha. \tag{5}$$

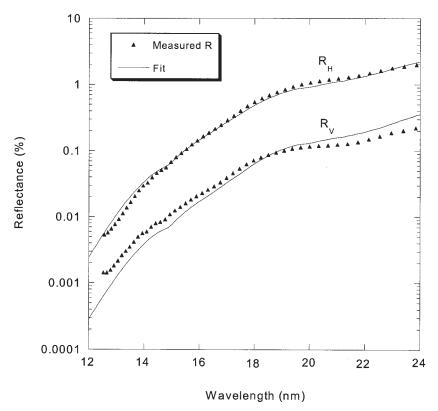


Fig. 2. Theoretical fits to the measured reflectances of the gold-coated mirror at an incident angle of 45° and in the wavelength region of 12–24 nm. The reflectometer is aligned vertically and horizontally for the measurements of R_V and R_H , respectively.

The reflectance polarization of a multilayer coating is defined as

$$P_{R} = (R_{S} - R_{P})/(R_{S} + R_{P})$$

$$= (R_{H} - R_{V})/[\alpha(R_{H} + R_{V})].$$
 (6)

Similar expressions can be obtained for the transmittance polarization, P_T , which is defined as

$$P_T = (T_S - T_P)/(T_S + T_P)$$

= $(T_H - T_V)/[\alpha(T_H + T_V)].$ (7)

The transmittance T is defined as the ratio of the transmitted intensity and the direct beam intensity. T_V and T_H are measured transmittances with the reflectometer oriented in vertical and horizontal directions, respectively. It should be noted that the transmitted intensity was measured by the underlying photodiode substrate, whereas the direct beam intensity was measured by the reference photodiode. There was a small difference between the responses of the two diodes, both measured before the multilayer depositions. This was taken into account when calculating the absolute transmittance value because the ratio between the responses of the two diodes throughout the whole measurement range is needed to normalize out such an effect. According to the polarization expressions, the transmittance polarization is negative if the *p*-polarized component is higher than the s-polarized component. Here the sign only represents a convention and is not a fundamental property.

4. Results and Discussion

The reflectance and transmittance of a Mo/B₄C/Si multilayer-coated photodiode were measured in the 12–24-nm wavelength region with the reflectometer aligned in the vertical (for R_V and T_V) and horizontal (for R_H and T_H) orientations, where incident radiation is mainly p-polarized and s-polarized, respectively. A freestanding silicon filter was used to suppress a small amount of residual higher-harmonic radiation from the monochromator. The wavelength scale was calibrated based on the silicon L_3 attenuation edge at a wavelength of 12.434 nm to an accuracy of 0.002 nm. 15 The angular motions of the sample and the detector were computer controlled to well within $\pm 0.1^{\circ}$ of accuracy. There was no observed difference between the reflectance results obtained from the witness sample and the silicon photodiode, implying that the surface roughness of the silicon wafer substrate was approximately the same as that of the silicon photodiode. Therefore only the results obtained from the photodiode are presented from here on.

The reflectances R_S and R_P of the Mo/B₄C/Si multilayer were calculated based on Eqs. (4) and (5), with a known α value of 0.89 and the measured R_V and R_H values. Similar calculations were performed for all transmittance signals. As shown in Fig. 4, the peak s-polarized reflectances (R_S) were 69.9% at 13.527

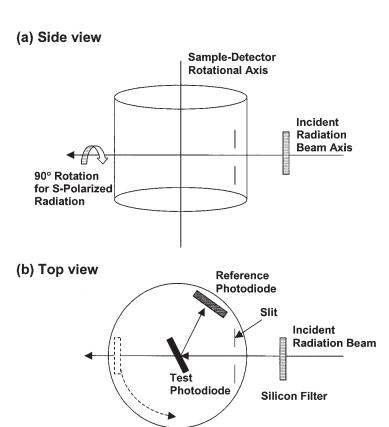


Fig. 3. (a) Layout of the reflectometer setup at the National Synchrotron Light Source beamline X24C for primarily p-polarized incident radiation, with the rotational axis of the sample–detector aligned in the vertical direction and the electric field vector parallel to the plane of incidence. (b) Cylindrical vacuum chamber rotated 90° about the incident beam axis so that the incident radiation is primarily s-polarized with the electric field normal to the plane of incidence. The reflectance and transmittance measurement can be performed at near-grazing to near-normal incident angles with the sample–detector rotated in the θ -2 θ configuration.

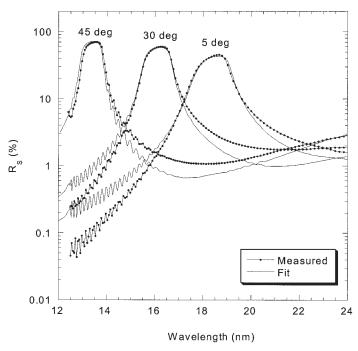


Fig. 4. Theoretical fits to the measured s-polarized reflectances of the $Mo/B_4C/Si$ multilayer (after taking into account the polarization of the incident beam) at angles of 5° , 30° , and 45° from normal incidence.

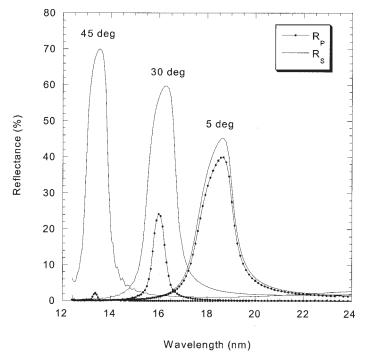


Fig. 5. Comparison of the measured s-polarized and p-polarized reflectances of the Mo/ B_4 C/Si multilayer at incident angles of 5°, 30°, and 45°.

nm, 59.5% at 16.277 nm, and 45.3% at 18.638 nm at 45°, 30°, and 5° from normal incidence, respectively. The structural parameters of the multilayer were determined by simultaneously fitting the s-polarized reflectance curves at the three incident angles. The best fits to these reflectance curves showed that the

 ${\rm Mo/B_4C/Si}$ multilayer had a 9.92-nm period thickness, with silicon, molybdenum, and boron carbide thicknesses of 6.38, 3.14, and 0.4 nm, respectively. In the fitting process approximately 2.5 nm of the last silicon layer was allowed to form a more stable compound of silicon dioxide, and the roughnesses of the

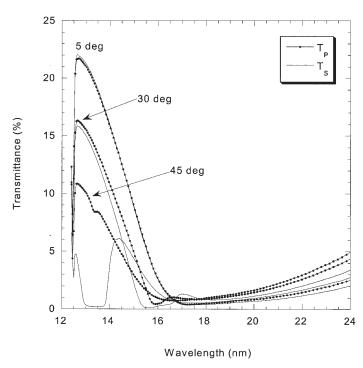


Fig. 6. Comparison of the measured s-polarized and p-polarized transmittances of the Mo/ B_4 C/Si multilayer at incident angles of 5°, 30°, and 45°.

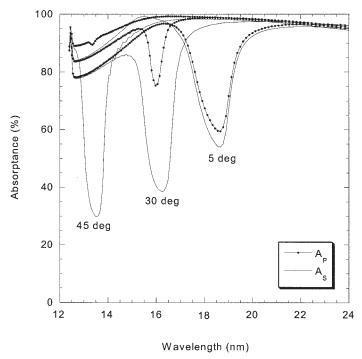


Fig. 7. Inferred values of the s-polarized and p-polarized absorptance based on the reflectances and transmittances of the Mo/B₄C/Si multilayer at incident angles of 5°, 30°, and 45°.

surface and all interfaces were assumed to be equal and were determined to be $\sim\!0.5$ nm. The above fitted values were, in fact, very similar to the designed values.

 R_S and R_P at three incident angles are compared in Fig. 5. It can be seen that R_S and R_P were almost

equal at near-normal incidence (5°). As the angle increased toward 45°, R_S increased while R_P decreased. The maximum separation between the two components occurred near 45°, where R_S was 69.9% at 13.527 nm and R_P was 2.4% at 13.361 nm. T_S and T_P at three incident angles are compared in Fig. 6,

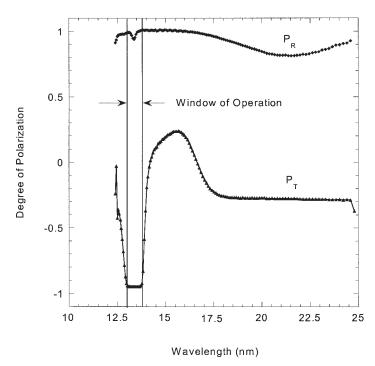


Fig. 8. Polarization performance of the $Mo/B_4C/Si$ multilayer through reflectance and transmittance measurements that are optimized at a 45° Brewster angle.

where T_S was very low (0.2%) in the 13.5-nm wavelength region at a 45° angle of incidence. Hence, the highest R_S value at an incident angle of 45° resulted in the lowest T_S value of 0.2%, and the lowest R_P value resulted in the highest T_P value of 8.4%. Based on the measured reflectance and transmittance information in each polarization, the absorptance was defined as $A_S = (1 - R_S - T_S)$ and $A_P = (1 - R_P - T_P)$, and the inferred values were plotted in Fig. 7. It can be seen that the enhancement of polarization near the Brewster angle was observed not only in the reflectance but also in the transmittance and absorptance spectra. This is a unique feature of the multilayer polarizer device. In addition, the maximum polarization occurred when the s-polarized component reached its minimum in transmittance and absorptance; this convention was opposite in reflectance. For these reasons, reflectance was the primary term for understanding the polarization performance: high s-polarized reflectance results in low s-polarized transmittance and absorptance and vice versa.

The polarization performance of the Mo/B₄C/Si multilayer obtained through reflectance and transmittance measurements (45°) is plotted in Fig. 8. A window of operation of approximately 0.75 nm wide (from the wavelength region of 13–13.7 nm) represented the region where the reflectance signal was sufficiently high for detection by the reference photodiode detector and where the transmittance signal was measurable by the underlying photodiode detector. The polarization of -0.95 was achieved with a Mo/B₄C/Si multilayer in transmittance, and a value of as high as 0.999 could also be achieved in reflectance. The change in sign of the transmittance polarization (P_T) from negative to positive was simply due to the T_S value's being higher than T_P in some wavelength region.

As seen in Fig. 8, the multilayer-coated photodiode device can function as a polarimeter in the traditional reflection manner over a broad wavelength range. In this case the polarimeter is composed of a reflector at 45° and a separate detector. In the transmission mode of operation, the multilayer-coated photodiode functions best as a polarimeter over a narrower wavelength range and can be used to study a spectral line or a group of closely spaced spectral lines. The benefit of transmission operation is that the device is monolithic, with integrated coating and detector features, and that the optical properties of the coating in transmission are less susceptible to surface contamination and oxidation that can significantly affect the traditional reflection mode of operation.

5. Conclusions

We have developed an EUV polarization sensitive detector using a $Mo/B_4C/Si$ multilayer as the polarizer. Because the multilayer was coated directly onto the photodiode substrate, its reflectance and transmittance could be measured simultaneously without the need of fragile freestanding samples. Instead of a standard Mo/Si multilayer, additional B_4C layers were introduced as barrier layers against

silicide formation and were applied at all Mo-on-Si interfaces to improve the reflectance performance of the multilayer. The Mo/B $_4$ C/Si multilayer was reflectance optimized near the Brewster angle, and it was found to function well as both reflectance and transmittance polarizers. The maximum polarizations obtained from transmittance and reflectance of the Mo/B $_4$ C/Si multilayer at a wavelength of approximately 13.5 nm and at an incident angle of 45° were as high as 95% and 99.9%, respectively. This result demonstrates the high polarization sensitivity and the usefulness of multilayer-coated photodiodes as EUV polarimeters.

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